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(54) METHOD FOR ORIENTING LIQUID CRYSTAL MOLECULE

(57)Abstract:

PROBLEM TO BE SOLVED: To orient liquid crystal molecules, while controlling the inclination angle of the orientation.

SOLUTION: This method for orienting the liquid crystals, comprising forming a liquid crystal layer comprising liquid crystal molecules on a substrate to orient the liquid crystal molecules, characterized by adding a pyridinium quaternary salt to the liquid crystal layer or the adjacent layer to control the inclination angle of the liquid crystal molecules by the action of the pyridinium quaternary salt.

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METHOD FOR THE ORIENTATION OF LIQUID CRYSTAL MOLECULES

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[There are no amendments to this patent.]

Abstract

Problem

Liquid crystal molecules are orientated while the orientation inclination angle is controlled.

Means to solve

A liquid crystal layer containing liquid crystal molecules is formed on a substrate, a pyridinium quaternary salt is added to the liquid crystal layer or a layer adjacent to it, and liquid crystal molecules are orientated while the inclination angle of liquid crystal molecules is controlled by the action of the pyridinium quaternary salt.

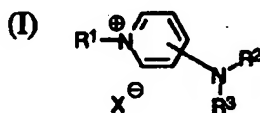
Claims

1. A method for the orientation of liquid crystal molecules characterized by the fact that, in a method for the formation of a liquid crystal layer containing liquid crystal molecules on a substrate and the orientation of liquid crystal molecules, a pyridinium quaternary salt is added to the liquid crystal layer or a layer adjacent to it, and the inclination angle of liquid crystal molecules is controlled by the action of the pyridinium quaternary salt.

2. The method for the orientation of liquid crystal molecules described in Claim 1, in which the pyridinium ring of the pyridinium quaternary salt has an aliphatic amino group as a substituent.

3. The method for the orientation of liquid crystal molecules described in Claim 1, in which the pyridinium quaternary salt is represented by the following formula (I):

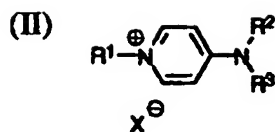
[Structure 1]



[in this formula, R¹ is an aliphatic group with 1 to 30 carbon atoms, R² and R³ are independently aliphatic groups with 1 to 8 carbon atoms, or R² and R³ are bonded to form a nitrogen-containing heterocycle; and X is an anion].

4. The method for the orientation of liquid crystal molecules described in Claim 1, in which the pyridinium quaternary salt is represented by the following formula (II):

[Structure 2]



[in this formula, R^1 is an aliphatic group with 1 to 30 carbon atoms, R^2 and R^3 are independently aliphatic groups with 1 to 8 carbon atoms, or R^2 and R^3 are bonded to form a nitrogen-containing heterocycle; and X is an anion].

5. The method for the orientation of liquid crystal molecules described in Claim 1, in which the pyridinium salt is used in the range of 1/1000 to 250/1000 in a molar ratio with respect to liquid crystal molecules.

6. The method for the orientation of liquid crystal molecules described in Claim 1, in which the liquid crystal molecules are discotic liquid crystal molecules.

7. The method for the orientation of liquid crystal molecules described in Claim 1, in which the liquid crystal molecules have triphenylene nuclei.

Detailed explanation of the invention

[0001]

Technical field of the invention

The present invention relates to a method for the orientation of liquid crystal molecules that is capable of controlling the orientation inclination angle.

[0002]

Prior art

Because a liquid crystal compound exhibits the combined properties of fluidity like a liquid and a regular molecular arrangement like a crystal on a liquid crystal layer, application development in a variety of fields has been expected. The control of its molecular arrangement has been indispensable in its engineering applications to liquid crystal devices represented by a liquid crystal display. In particular, the control of the inclination angle beginning with the pretilt angle has been one of the important tasks in the liquid crystal orientation technology.

[0003]

There are seven known typical molecular orientations of liquid crystals: 1) homeotropic orientation, 2) homogeneous orientation, 3) tilt orientation, 4) hybrid orientation, 5) twisted orientation, 6) planar orientation, and 7) focal conic orientation. Details are described in

Fundamentals and Applications of Liquid Crystals, published by Industrial Survey Association (1991).

[0004]

In general, the type of orientation to be obtained can be decided by the type of orientation treatment chosen to be conducted on the substrate surface of the liquid crystal. A variety of methods have been proposed for the orientation treatment. These are described in detail in Fundamentals and Applications of Liquid Crystals, published by Industrial Survey Association (1991). For example, as methods for perpendicular or parallel orientation, methods for the chemical adsorption of a chromium carboxylate complex, an organic silane or other orientating agent on the substrate surface are disclosed in Appl. Phys. Lett. J., Vol. 27, p. 268 (1975), Appl. Phys. Lett. J., Vol. 29, p. 67 (1976), Appl. Phys. Lett. J., Vol. 22, p. 111 (1973), etc.; methods for the physical adsorption of orientating agents on the substrate surface in Oyo Butsuri Shi, Vol. 43, p. 18 (1974), Phys. Rev. Lett. J., Vol. 25, p. 67 (1976), etc.; methods for the polymerization adherence of a low molecular weight substance on the substrate surface by plasma discharge in Appl. Phys. Lett. J., Vol. 24, p. 297 (1974), etc.; and methods for the polymerization adherence of a high molecular weight substance on the substrate surface by the action of a high electric field in J. Appl. Phys. Lett., Vol. 47, p. 1270 (1976), etc. Next, as methods for inclined parallel orientation, inclined vapor deposition methods by the vapor deposition of silicon oxide or other oxides on the substrate surface from an inclined angle are disclosed in Appl. Phys. Lett. J., Vol. 25, p. 479 (1974), etc.. As methods for inclined perpendicular orientation, methods using the previously described inclined vapor deposition methods and perpendicular orientating agents in combination are disclosed. As a method for inclined perpendicular orientation, the inclined vapor deposition method for the vapor deposition of silicon oxide or other oxides from an inclined angle while the substrate surface is rotated is disclosed in the 6th Liquid Crystal Seminar Abstracts, p. 96 (1980). With this method, problems occur in which productivity is poor, area enlargement is difficult, etc., in the industrial manufacture of a product with stabilized orientation having a constant inclination orientation angle.

[0005]

As a method for the orientation of molecules, there is a method in which an organic film is coated on the substrate surface, the surface is rubbed with cloth of cotton, nylon, polyester or the like in a constant direction, and liquid crystal molecules are orientated in the rubbing direction. Since a stable orientation can be obtained relatively easily by this method, it is solely used in industry. As organic films, polyvinyl alcohol, polyoxyethylene, polyamide, polyimide, etc., can be mentioned. Polyimide is most commonly used from aspects of chemical stability,

thermal stability, etc. For the liquid crystal orientation films like this, it is known that the inclination angle of liquid crystal molecules is changed by the orientation film used. A variety of orientation films capable of inclined orientation are disclosed in, for example, Japanese Kokai Patent Application Nos. Hei 5[1993]-43687, Hei 8[1996]-12759, Hei 8[1996]-220541, Hei 8[1996]-220542, Hei 9[1997]-143196, Hei 9[1997]-230354, Hei 9[1997]-278724, Hei 10[1998]-45690, and Hei 10[1998]-123532. However, the inclination angle depends on the properties of the orientation film itself. It has been impossible to adjust the angle easily. A method for the control of the orientation of liquid crystal molecules with light is also known. For example, in Kino Zairyo, Vol. 17, p. 13 (1997), a variety of orientation films capable of controlling the inclination angle with light are described. However, with these methods, it is necessary to use polarized light irradiation or polarized light ultraviolet ray irradiation. It is necessary to change the angle of incidence of polarized light in the adjustment of the inclination angle. The inclination angle cannot be controlled easily.

[0006]

A variety of orientation technologies like those described previously have been proposed, and technologies for inclined orientation of liquid crystal compounds have also been disclosed. However, from the viewpoint of control of the inclination angle, none of them is sufficient. It is desirable to develop a method capable of controlling the arrangement [sic; orientation] inclination angle of liquid crystal compounds in an easy manner.

[0007]

Problems to be solved by the invention

The task of the present invention is to easily control the orientation inclination angle of liquid crystal compounds.

[0008]

Means to solve the problems

The present invention provides a method for the orientation of liquid crystal molecules by (1) through (4) in the following.

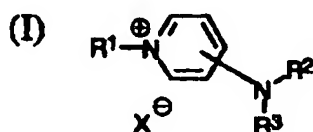
(1) A method for the orientation of liquid crystal molecules characterized by the fact that, in a method for the formation of a liquid crystal layer containing liquid crystal molecules on a substrate and the orientation of liquid crystal molecules, a pyridinium quaternary salt is added to the liquid crystal layer or a layer adjacent to it, and the inclination angle of liquid crystal molecules is controlled by the action of the pyridinium quaternary salt.

(2) The method for the orientation of liquid crystal molecules described in Claim [sic] 1, in which the pyridinium ring of the pyridinium quaternary salt has an aliphatic amino group as a substituent.

(3) The method for the orientation of liquid crystal molecules described in (1), in which the pyridinium quaternary salt is represented by the following formula (I):

[0009]

[Structure 3]



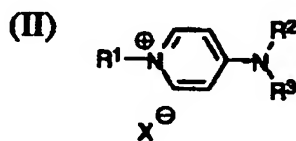
[0010]

[in this formula, R^1 is an aliphatic group with 1 to 30 carbon atoms, R^2 and R^3 are independently aliphatic groups with 1 to 8 carbon atoms, or R^2 and R^3 are bonded to form a nitrogen-containing heterocycle; and X is an anion].

(4) The method for the orientation of liquid crystal molecules described in (1), in which the pyridinium quaternary salt is represented by the following formula (II):

[0011]

[Structure 4]



[0012]

[in this formula, R^1 is an aliphatic group with 1 to 30 carbon atoms, R^2 and R^3 are independently aliphatic groups with 1 to 8 carbon atoms, or R^2 and R^3 are bonded to form a nitrogen-containing heterocycle; and X is an anion].

(5) The method for the orientation of liquid crystal molecules described in (1), in which the pyridinium salt is used in the range of 1/1000 to 250/1000 in a molar ratio with respect to liquid crystal molecules.

(6) The method for the orientation of liquid crystal molecules described in (1), in which the liquid crystal molecules are discotic liquid crystal molecules.

(7) The method for the orientation of liquid crystal molecules described in (1), in which the liquid crystal molecules have triphenylene nuclei.

[0013]

Embodiments of the invention

In this specifications document, the substrate represents a material for loading a liquid crystal layer, or a material on which to place a liquid crystal layer as a constituent element of liquid crystal cells. Specifically, polymer films like triacetylcellulose (TAC) films and polyethylene naphthalate (PEN) films are used. Glass plates, ITO substrates, color filter substrates, quartz plates, silicon wafers, and polarized light plates can also be used as substrates. On the substrate, an orientation layer, transparent electrode (ITO), color filter layer, black matrix, and a thin film transistor can also be provided. Electrode pattern formation, a rubbing treatment such as that described previously, polarized light UV irradiation, or other orientation treatments can also be carried out. The substrate can be used alone or as a pair. In the case of usage as a pair, if necessary, a spacer, a sealing agent or the like can also be used. In this specifications document, it is preferable that the layer adjacent to the liquid crystal layer is the layer nearest the liquid crystal layer among the layers located between the substrate and the liquid crystal layer. It is also acceptable that the layer adjacent to the liquid crystal layer functions as an orientation film or a transparent electrode.

[0014]

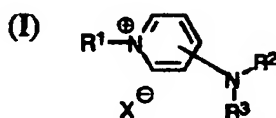
The liquid crystal layer is constituted mainly by liquid crystal molecules. As the liquid crystal molecules, discotic liquid crystal molecules, rod-shaped liquid crystal molecules, and cholesteric liquid crystal molecules are preferred. Discotic liquid crystal molecules are especially preferred. It is preferable that the discotic liquid crystal molecules have triphenylene nuclei. Two or more types of liquid crystal molecules can also be used in combination. Components (such as a colorant, dichroic colorant, polymer, polymerizing agent, sensitizing agent, phase transition temperature depressant, and stabilizer) can also be added to the liquid crystal layer in addition to the liquid crystal molecules. As methods for applying the liquid crystal layer to the substrate, publicly known methods can be adopted. As coating types, publicly known methods, for example, the curtain coating method, extrusion coating method, roll coating method, spin coating method, dip coating method, bar coating method, spray coating method, printing coating method, etc., can be used. At this time, on the substrate, the necessary treatments described previously, the necessary layers starting with the orientation layer, and parts can also be applied. As methods for the injection of liquid crystal molecules between substrates, the dispenser mode, bell-jar method and other ordinary methods can be used. It is also acceptable that a liquid crystal layer is coated on a substrate and used in combination with another substrate or a substrate coated with another liquid crystal layer.

[0015]

In the present invention, a pyridinium quaternary salt is used and the inclination angle of liquid crystal molecules is controlled by its action. If a pyridinium quaternary salt is added, in general, the inclination angle of liquid crystal molecules is increased. As a result, the inclination angle of liquid crystal molecules can be controlled. It is preferable that the pyridinium ring of the pyridinium quaternary salt has an aliphatic amino group as a substituent. It is preferable that the substitution position of the aliphatic amino group is position 4. The pyridinium ring may also have other substituents (amino, hydroxyl, thiol, carboxyl, an alkyl group, an alkoxy group, or a halogen atom) in addition to the aliphatic amino group. It is preferable that the N-substituent (the group bonded to a nitrogen atom) of the pyridinium quaternary salt is an aliphatic group. The aliphatic groups include alkyl groups, substituted alkyl groups, alkenyl groups, substituted alkenyl groups, alkynyl groups, and substituted alkynyl groups. The aliphatic group may also have a cyclic structure. A linear aliphatic group may also have branches. Examples of substituents of the aliphatic group include aryl groups, alkoxy groups, alkylthio groups, amido groups, acyl groups, acyloxy groups, alkoxycarbonyl groups, carboxyls, halogen atoms, alkoxyalkyl groups, alkyloxyoxycarbonyl alkyl groups, and 2,2,3,3,4,4,5,5,6,6,7,7,8,8,8-pentadecafluorooctyloxycarbonyl ethyl. Examples of anions as counterions of the pyridinium quaternary salt include fluorine ions, chlorine ions, bromine ions, iodine ions, benzene sulfonium ions, and p-toluene sulfonium ions. The pyridinium quaternary salt represented by the following formula (I) is preferred.

[0016]

[Structure 5]



[0017]

In the formula (I), R^1 is an aliphatic group with 1 to 30 carbon atoms. The aliphatic groups include alkyl groups, substituted alkyl groups, alkenyl groups, substituted alkenyl groups, alkynyl groups, and substituted alkynyl groups. The aliphatic group may also have a cyclic structure. A cyclic aliphatic group may contain a group having a steroid structure. A linear aliphatic group may also have branches. Examples of substituents of the aliphatic group include aryl groups, alkoxy groups, alkylthio groups, amido groups, acyl groups, acyloxy groups, alkoxycarbonyl groups, carboxyls, halogen atoms, alkoxy alkyl groups, alkyloxyoxycarbonylalkyl groups, and 2,2,3,3,4,4,5,5,6,6,7,7,8,8,8-pentadecafluorooctyloxycarbonyl ethyl are included.

[0018]

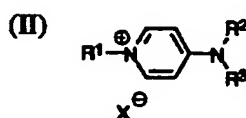
In the formula (I), R^2 and R^3 are independently aliphatic groups with 1 to 8 carbon atoms, or R^2 and R^3 are bonded to form a nitrogen-containing heterocycle. The aliphatic groups include alkyl groups, substituted alkyl groups, alkenyl groups, substituted alkenyl groups, alkynyl groups, and substituted alkynyl groups. Linear aliphatic groups are preferred over cyclic aliphatic groups. It is preferable that R^2 and R^3 are independently alkyl groups, substituted alkyl groups, alkenyl groups, or substituted alkenyl groups. It is further preferable that they are alkyl groups or substituted alkyl groups. It is most preferable that they are alkyl groups. It is preferable that the number of carbon atoms of R^2 and R^3 is 1 to 7. It is further preferable that it is 1 to 5. It is most preferable that it is 1 to 4. Examples of aliphatic groups include methyl, ethyl, n-propyl, isopropyl, cyclopropyl, n-butyl, sec-butyl, t-butyl group, cyclobutyl group, cyclopropylmethyl, n-pentyl, neopentyl, n-hexyl, cyclohexyl, and n-heptyl. It is preferable that the nitrogen-containing heterocycle formed by bonding of R^2 and R^3 is a 5-member ring or a 6-member ring.

[0019]

In the formula (I), X is an anion. Examples of anions include chlorine ions, bromine ions, iodine ions, p-toluene sulfonium ions, and benzene sulfonium ions. The pyridinium quaternary salt represented by the following formula (II) is further preferred.

[0020]

[Structure 6]

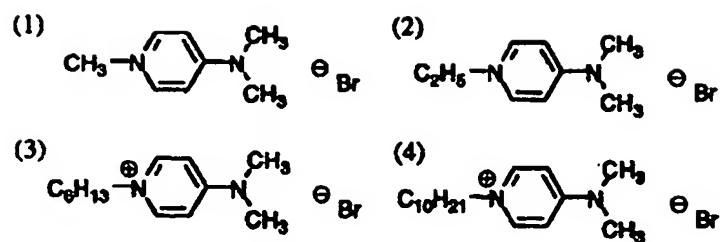


[0021]

In the formula (II), R^1 , R^2 , R^3 and X have the same definitions as in the formula (I). Examples of the pyridinium quaternary salt are shown in the following.

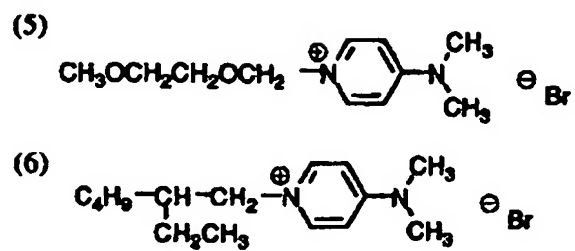
[0022]

[Structure 7]



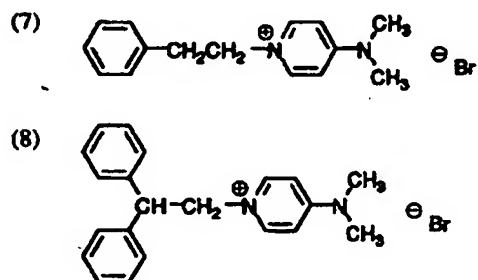
[0023]

[Structure 8]



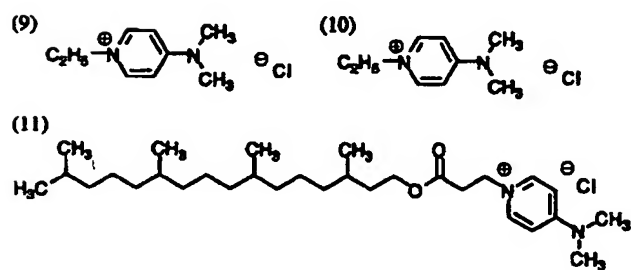
[0024]

[Structure 9]



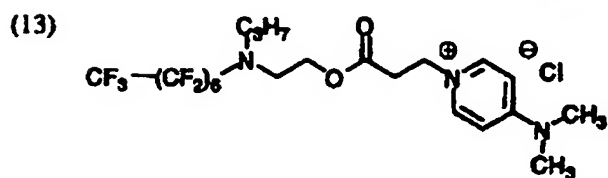
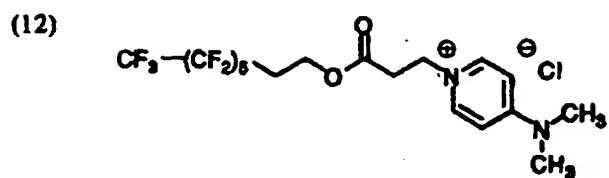
[0025]

[Structure 10]



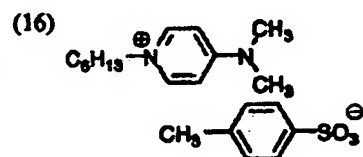
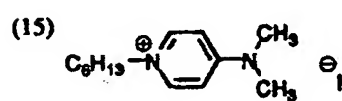
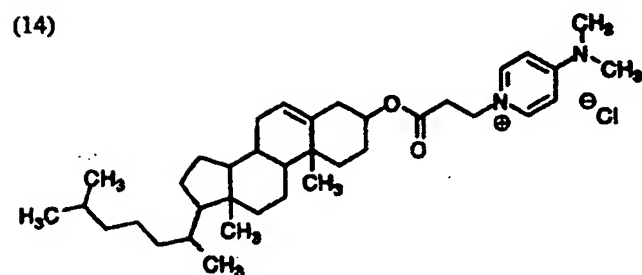
[0026]

[Structure 11]



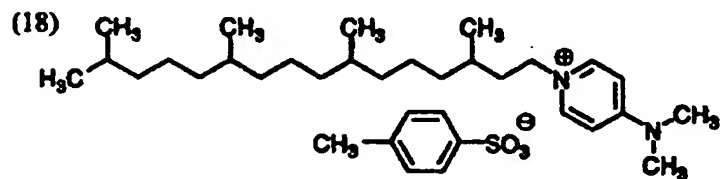
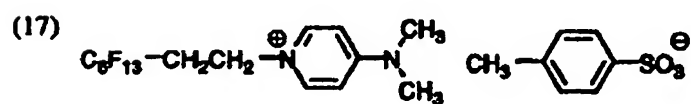
[0027]

[Structure 12]



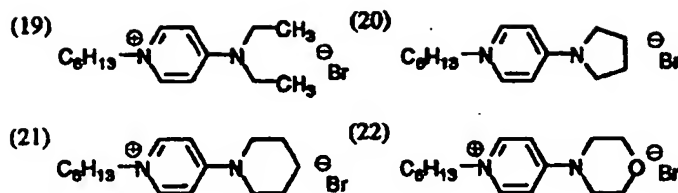
[0028]

[Structure 13]



[0029]

[Structure 14]



[0030]

As examples of methods for the addition of a pyridinium quaternary salt into a layer adjacent to the liquid crystal layer, it is possible to mention a method for the addition of a pyridinium quaternary salt into a coating solution in order to form a layer adjacent to the liquid crystal layer on a substrate, a method for the direct coating of a solution of a pyridinium quaternary salt as a layer adjacent to the liquid crystal layer, etc. However, the method for the addition of a pyridinium quaternary salt into a coating solution in order to form a layer adjacent to the liquid crystal layer is preferred. In regard to the amount of addition, an amount appropriate to the desired inclination angle in a range without disturbing the orientation of the liquid crystal can be adopted. The addition in the range of 1/1000 to 250/1000 in a molar ratio with respect to liquid crystal molecules used can be adopted as a preferred example. As coating modes, examples of the coating modes for the liquid crystal layer described previously can be mentioned. As an example for the addition of a pyridinium quaternary salt into a liquid crystal layer itself, it is possible to adopt a method for the addition of a pyridinium quaternary salt or a solution containing said quaternary salt after the coating formation of the liquid crystal layer on the substrate, a method for the addition of a pyridinium quaternary salt or a solution containing said quaternary salt after the injection of liquid crystal molecules between a pair of substrates, or the like.

[0031]

As an example for the injection of liquid crystal molecules between substrates, it is possible to add a pyridinium quaternary salt at an amount appropriate for the desired inclination angle in a range without disturbing the orientation of the liquid crystal into the injection solution of the liquid crystal manufactured by an ordinary method. At this time, another additive can also be used in combination. There are no special restrictions on the amount of addition as long as it is in a range without disturbing the orientation of the liquid crystal. The addition in the range of 1/1000 to 25/100 in a molar ratio with respect to liquid crystal molecules used can be used as a preferred example. As a method for the addition of liquid crystal molecules into a solution to be coated on the substrate, it is possible to add a pyridinium quaternary salt at an amount

appropriate for the desired inclination angle in a range without disturbing the orientation of the liquid crystal manufactured by an ordinary method. At this time, another additive can also be used in combination. There are no special restrictions on the amount of addition as long as it is in a range without disturbing the orientation of the liquid crystal. The addition in the range of 1/1000 to 25/100 in a molar ratio with respect to liquid crystal molecules used can be used as a preferred example.

[0032]

Application examples

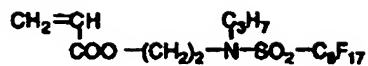
Application Example 1

(Evaluation of liquid crystal inclination angle controlling capability) An aqueous solution of polyvinyl alcohol modified with methacryloyloxyisocyanate described in Japanese Kokai Patent Application No. Hei 8[1996]-48197 was coated on a glass substrate (thickness: 0.85 mm) with a bar coater, dried, and subjected to a rubbing treatment. Next, a solution obtained by the dissolution of 100 parts by weight of a discotic liquid crystal compound (DLC) described in the following, 1 part by weight of cellulose acetate butyrate, and 0.05 part by weight of a pyridinium quaternary salt in 400 parts by weight of methyl ethyl ketone was coated with a spin coater and dried at room temperature. After heating the liquid crystal layer to 200°C, the liquid crystal compound was orientated at 130°. The substrate in such a state was rapidly cooled to room temperature to fix the orientation state. By measurement of the angle in a direction such that the retardation of the thin film obtained was minimum, the average inclination angle of its liquid crystal molecules was calculated.

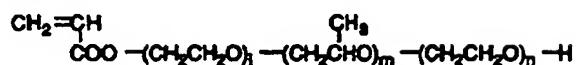
[0036]

[Structure 16]

M-1



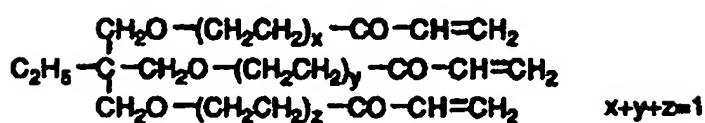
M-2



[0037]

[Structure 17]

M-3



[0038]

① 使用傾斜制御剤 未使用④	② 添加量 (対液晶モル比)	③ 平均傾斜角
化合物 1	0.3	33度
化合物 2	0.3	36度
化合物 3	0.3	36度
化合物 4	0.3	37度
化合物 5	0.3	37度
化合物 6	0.3	35度
化合物 7	0.3	37度
化合物 8	0.3	35度
化合物 9	0.3	37度
化合物 10	0.3	36度
化合物 11	0.3	37度
化合物 12	0.3	36度
化合物 13	0.3	35度
化合物 14	0.3	37度
化合物 15	0.3	38度
化合物 16	0.3	38度
化合物 17	0.3	37度
化合物 18	0.3	36度
化合物 19	0.3	36度
化合物 20	0.3	38度
化合物 21	0.3	38度

- Key: 1 Inclination controlling agent used
 2 Amount added (molar ratio with respect to liquid crystals)
 3 Average inclination angle
 4 Not used
 5 — degrees

6 Compound ____

[0039]

Application Example 3

(Evaluation of liquid crystal inclination angle controlling capability) Sunever [transliteration] SE-610 (manufactured by Nissan Chemical Co., Ltd.) was coated as an orientation film on a glass substrate with a bar coater (using No. 4 bar), dried, and subjected to a rubbing treatment. Next, a solution obtained by the dissolution of the discotic liquid crystal compound (100 parts by weight) used in Application Example 1, cellulose acetate butyrate (1 part by weight), and compound 3 (0.5 molar ratio with respect to liquid crystals) into methyl ethyl ketone (400 parts by weight) was coated with a spin coater and dried at room temperature. After heating of the liquid crystal layer to 200°C, the liquid crystal compound was orientated at 130°. The substrate in such a state was rapidly cooled to room temperature to fix the orientation state. Based on observation of the liquid crystal compound and the crystal rotational method, it was confirmed that the discotic liquid crystal compound was arranged in parallel with respect to the rubbing direction, and the orientation was perpendicular (homogeneous) with respect to the substrate and uniform (monodomain). On the other hand, in a system as the control without the addition of the pyridinium quaternary salt, no uniform orientation state was obtained.

[0040]

Application Example 4

(Evaluation of liquid crystal inclination angle controlling capability) Sunever SE-610 (manufactured by Nissan Chemical Co., Ltd.) was coated as an orientation film on a glass substrate with a bar coater (using No. 4 bar), dried, and subjected to a rubbing treatment. Next, a solution obtained by the dissolution of the discotic liquid crystal compound (100 parts by weight) used in Application Example 1 and compound 3 (0.5 molar ratio with respect to liquid crystals) in methyl ethyl ketone (400 parts by weight) was coated with a spin coater and dried at room temperature. On the liquid crystal layer coated-substrate obtained, another glass substrate obtained by coating Sunever SE-610 as an orientation film on a glass substrate with a bar coater, drying, and carrying out a rubbing treatment was rotated by 180° in the rubbing direction, and matched. Next, after heating of the liquid crystal layer to 200°C, the liquid crystal compound was orientated at 130°. The substrate in such a state was rapidly cooled to room temperature to fix the orientation state. By observation with a polarized microscope, the arrangement of liquid crystal molecules in the rubbing direction and inclined orientation with respect to the substrate were confirmed. The inclination angle was measured based on the crystal rotational method.

[0041]

Inclination controlling agent used	Average inclination angle
Not used	32 degrees
Compound 3	40 degrees

[0042]

Application Example 5

(Evaluation of liquid crystal inclination angle controlling capability) The relationship between the amount of addition and the inclination angle was investigated in the same manner as in Application Example 2 by using compound (2) and compound (3). As shown in the following, a concentration-dependent inclination angle rise was confirmed.

[0043]

① 使用傾斜制御剤	② 添加量	③ 平均傾斜角
未使用 ④	—	33度
⑥ 化合物2	⑦ { 0.05重量部	36.2度
	0.10重量部	37.1度
	0.15重量部	38.0度

Key: 1 Inclination controlling agent used
 2 Amount added
 3 Average inclination angle
 4 Not used
 5 ___ degrees
 6 Compound ___
 7 part by weight

[0044]

化合物3	⑦ { 0.05重量部	36.3度
⑥	0.10重量部	38.5度
	0.15重量部	39.6度

Key: 5 ___ degrees
 6 Compound ___
 7 part by weight

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